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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/EP97/00896 <b>(22) International Filing Date:</b> 21 February 1997 (21.02.97) <b>(30) Priority Data:</b> 08/670,887 26 June 1996 (26.06.96) US <b>(71) Applicant (for AU BB CA GB IE IL KE LC LK LS MN MW NZ SD SG SZ TT UG only):</b> UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB). <b>(71) Applicant (for all designated States except AU BB CA GB IE IL KE LC LK LS MN MW NZ SD SG SZ TT UG):</b> UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). <b>(72) Inventors:</b> FARRELL, Terence, James; 6613 McKinley Place, West New York, NJ 07093 (US). SCHAFER, Georgia; 15 Ridge Road, Rutherford, NJ 07070 (US). DALTON, James; 245 Lawton Avenue, Cliffside Park, NJ 07010 (US). <b>(74) Agent:</b> MOLE, Peter, Geoffrey; Unilever plc, Patent Division, Colworth House, Sharnbrook, Bedford MK44 1LQ (GB).		<b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> ADDITIVE COMPOSITION FOR DELIVERING BENEFIT AGENT AND CLEANSING BARS CONTAINING SAID ADDITIVES  <b>(57) Abstract</b>  The present invention relates to novel chip compositions comprising alkylene glycol, benefit agent and fumed silica. Use of chips comprising excess of alkylene glycol over benefit agent and fumed silica has been found to allow significant deposition of benefit agent without compromising processing. In a second embodiment, the invention comprises bar compositions comprising mixtures of the chips of the invention and chips containing defined surfactant systems. The invention further comprises a process for forming the chips of the invention and a method of enhancing deposition without compromising processing using the chips of the invention.		

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**ADDITIVE COMPOSITION FOR DELIVERING BENEFIT AGENT AND CLEANSING BARS CONTAINING SAID ADDITIVES**FIELD OF THE INVENTION

5 The present invention relates to bar compositions, particularly synthetic soap bar compositions, able to deliver beneficial agents (e.g., silicone) in higher amounts than previously possible. In particular, the invention relates to  
10 additive compositions (e.g., soap chips) comprising a benefit agent which are then mixed with non-beneficial agent chips prior to milling, extruding and stamping the bars. The invention further relates to a method of making the additives.

BACKGROUND OF THE INVENTION

15 It has long been a desirable goal to deliver some kind of benefit agent (e.g., silicone or other oils) to the skin through a personal wash composition.

20 In liquid cleansers, for example, cationic hydrophilic polymers such as Polymer JR<sup>(R)</sup> from Amerchol or Jaguar<sup>(R)</sup> from Rhone Poulenc have been used to enhance delivery of benefit agents (EP 93,602; WO 94/03152; and WO 94/03151). In  
25 applicants' copending application, U.S. Serial No. 08/412,803 to Tsaur et al., separate hydrogel particles act as a structure to entrap the benefit agent in concentrated form.

30 Delivery of benefit agents (e.g., silicone) in bar compositions has proven much more difficult for a number of reasons. If the benefit agent is soluble in any component of the bar composition, for example, it may simply solubilize into these components during the batch mixing phase (prior  
35 to cooling and chip formulation) and either no benefit agent

or an insufficient amount of benefit agent will be present in the final bar (after milling, plodding and extrusion of chips) to be delivered to the skin. The overly soluble benefit agent may also reduce viscosity and cause improper mixing. If the benefit agent is too viscous, on the other hand, it tends to get in the processing equipment and become too difficult to process.

U.S. patent No. 5,154,849 to Visscher et al. teaches bar compositions containing a silicone skin mildness/moisturizing aid component. In one embodiment, the silicone component may be mixed with a carrier which is selected to facilitate incorporation of the silicone. Preferred carrier is said to be polyethylene glycol. At column 16, the reference describes that silicone is mixed into melted Carbowax (polyethylene glycol), that the mixture is cooled to form flakes, and that the flakes are preferably added to an amalgamator.

It is clear, however, that the Visscher et al. reference contemplates a silicone/carrier system different from the benefit agent/carrier/fumed silica system of the subject invention. First, the Visscher patent does not teach fumed silica, a critical component of the additive compositions and one which is believed to provide the structure required to retain and engulf the benefit agent (e.g., silicone). Second, as suggested above and as shown in Figures 1 and 2, the structure of the carrier/silicone chip is distinct. The Visscher et al. chip does not contain the silicone in discrete droplets, but rather the silicone oozes and surrounds the carrier. By contrast, the benefit agent droplets of the invention are discrete droplets retained within the chip. This helps to ensure the silicone does not ooze and interfere with processing.

The discrete particles of the invention, in turn, are present for two reasons, it is believed. The first, as noted above, is presence of fumed silica which, while not wishing to be bound by theory, it is believed helps to work with the carrier (i.e., PEG) to better entrap the silicone. The second is that, unlike the Visscher et al. system, the present invention requires there be an equal amount or more of carrier relative to the benefit agent. By contrast, it appears from Visscher et al., where eleven pounds of silicone (column 15, lines 1-2), are mixed with 5 to 6 pounds of Carbowax (column 15, line 29) that there is probably an excess of silicone to PEG and, at the least, there is no recognition of the criticality of having an equal amount or more of PEG to silicone.

In short, the chips of the Visscher reference are extremely difficult to process both because there is no control over the amount of silicone used and because there is no use of fumed silica.

#### SUMMARY OF THE INVENTION

Unexpectedly, applicants have found that, when specific additives are made containing an equal amount or greater of polyalkylene glycol to benefit agent and further containing fumed silica, the benefit agent (e.g., silicone) is prepared in the form of discrete droplets which in turn allow the benefit agent to be much more readily processed.

Specifically, the invention comprises a chip composition comprising:

- (a) 40% to about 80% by wt. of the chip composition of a polyalkylene glycol having a molecular weight greater than about 4,000, preferably 5,000 to 20,000, more preferably 5,000 to 10,000;

- (b) 10% to 40% by wt. of the chip composition of benefit agent (e.g., silicone);
- (c) 0.01% to 10% by wt. chip composition fumed silica;
- (d) 0.0% to 10% by wt. chip composition, preferably 0% to 5% by wt. water; and
- (e) 0% to 15% by wt. chip composition C<sub>8</sub> to C<sub>22</sub> fatty acid.

In a second embodiment of the invention, the invention comprises an extruded bar composition produced using about 20% to 40% chips as described above and about 80% to 60% chips comprising about 5% to 90% by wt. of a surfactant system wherein the surfactant is selected from the group consisting of soap, anionic surfactant, nonionic surfactant, amphoteric surfactant, zwitterionic surfactant, cationic surfactant and mixtures thereof. The "soap and/or surfactant" chips additionally may comprise other components typically found in such chips such as, for example, minor amounts of fragrance, preservative (e.g., butylated hydroxy toluene) skin feel polymer (e.g., guar) etc.

Although the surfactant system of the second (non-additive containing) chip may be a pure soap surfactant system, preferably the surfactant system comprises:

- (a) a first synthetic surfactant which is an anionic surfactant; and
- (b) a second synthetic surfactant selected from the group consisting of a second anionic different from the first, a nonionic, an amphoteric and mixtures thereof.

A particularly preferred surfactant system comprises acyl isethionate as the first anionic and a sulfosuccinate or a betaine surfactant or mixtures of the two.

In a third embodiment of the invention, the invention comprises a method of making benefit agent containing chips comprising:

- (a) 40% to 80% polyalkylene glycol;
- (b) 10% to 40% benefit agent;
- (c) 0.01% to 10% fumed silica;
- (d) 0% to 10% water; and
- (e) 0% to 10% C<sub>8</sub> to C<sub>22</sub> fatty acid; which method

comprises mixing the ingredients at temperatures above the melting point of polyalkylene glycol (i.e., above about 50°C) for 1 to 60 minutes; cooling on a chill roll (at about 0° to 25°C); and collecting.

#### BRIEF DESCRIPTION OF FIGURES

Figure 1 is a micrograph of a chip produced following process described in U.S. patent No. 5,154,849 to Visscher et al. wherein the chip is flooded with water and photographed under a microscope. The silicone does not form discrete particles but forms a large layer of silicone surrounding the polyalkylene glycol. The viscous silicone gets caught in machinery and inhibits processing.

Figure 2 is a micrograph of a chip product according to the subject invention. As noted, the benefit agent is found in discrete drops. Further, it is obvious, there is far less benefit agent present (e.g., to interfere with processing) than in Figure 1.

#### DETAILED DESCRIPTION OF THE INVENTION

In one embodiment of the invention, the present invention relates to novel soap chip compositions (e.g., in the process for making bars, molten compositions are formed which are then cooled on what is commonly called a chill roll

to form flakes or chips; these chips are subsequently refined and/or plodded to form billets which are stamped and cut to form final bars) which are readily processable in conventional soap machinery while still showing significant benefit agent deposition (i.e., comparable to deposition obtained in liquid body washes).

As seen from Figures 1 and 2, by carefully controlling the level of benefit agent (so that it cannot exceed the level of alkylene glycol carrier) and by utilizing fumed silica (while not wishing to be bound by theory, it is believed the fumed silica forms three dimensional networks, altering flow properties of, for example, silicone, and causes thickening), applicants have been able to provide discrete droplets of benefit agent so that the agent is unable to stick to the machinery and significantly inhibits processing.

#### CHIP COMPOSITION

##### Polyalkylene Glycol

The first component of the chip composition is the polyalkylene glycol carrier. This carrier should comprise about 40% to 80% by wt., preferably about 50% to 70% by wt. of the chip composition. Preferably, the polyalkylene glycol should have a molecular weight greater than 4,000 to about 100,000, preferably 4,000 to 10,000. An especially preferred carrier is polyethylene glycol, for example Carbowax PEG 8000<sup>(R)</sup> from Union Carbide.

##### Benefit Agent

The benefit agent "composition" of the subject invention may be a single benefit agent component or it may be a

benefit agent compound added via a carrier. Further the benefit agent composition may be a mixture of two or more compounds one or all of which may have a beneficial aspect. In addition, the benefit agent itself may act as a carrier for other components one may wish to add to the bar composition.

The benefit agent can be an "emollient oil" by which is meant a substance which softens the skin (stratum corneum) by increasing into water content and keeping it soft by retarding decrease of water content.

Preferred emollients include:

- (a) silicone oils, gums and modifications thereof such as linear and cyclic polydimethylsiloxanes; amino, alkyl alkylaryl and aryl silicone oils;
- (b) fats and oils including natural fats and oils such as jojoba, soybean, rice bran, avocado, almond, olive, sesame, persic, castor, coconut, mink oils; cacao fat; beef tallow, lard; hardened oils obtained by hydrogenating the aforementioned oils; and synthetic mono, di and triglycerides such as myristic acid glyceride and 2-ethylhexanoic acid glyceride;
- (c) waxes such as carnauba, spermaceti, beeswax, lanolin and derivatives thereof;
- (d) hydrophobic plant extracts;
- (e) hydrocarbons such as liquid paraffins, vaseline, microcrystalline wax, ceresin, squalene, pristan and mineral oil;
- (f) higher fatty acids such as lauric, myristic, palmitic, stearic, behenic, oleic, linoleic, linolenic, lanolic, isostearic and poly unsaturated fatty acids (PUFA);
- (g) higher alcohols such as lauryl, cetyl, stearyl,

oleyl, behenyl, cholesterol and 2-hexydecanol alcohol;

- 5 (h) esters such as cetyl octanoate, myristyl lactate, cetyl lactate, isopropyl myristate, myristyl myristate, isopropyl palmitate, isopropyl adipate, butyl stearate, decyl oleate, cholesterol isostearate, glycerol monostearate, glycerol distearate, glycerol tristearate, alkyl lactate, alkyl citrate and alkyl tartrate;
- 10 (i) essential oils such as mentha, jasmine, camphor, white cedar, bitter orange peel, ryu, turpentine, cinnamon, bergamot, citrus unshiu, calamus, pine, lavender, bay, clove, hiba, eucalyptus, lemon, starflower, thyme, peppermint, rose, sage, menthol,
- 15 cineole, eugenol, citral, citronelle, borneol, linalool, geraniol, evening primrose, camphor, thymol, spirantol, penene, limonene and terpenoid oils;
- 20 (j) lipids such as cholesterol, ceramides, sucrose esters and pseudo-ceramides as described in European Patent Specification No. 556,957;
- (k) vitamins such as vitamin A and E, and vitamin alkyl esters, including those vitamin C alkyl esters;
- 25 (l) sunscreens such as octyl methoxyl cinnamate (Parsol MCX) and butyl methoxy benzoylmethane (Parsol 1789);
- (m) phospholipids; and
- (n) mixtures of any of the foregoing components.

30 A particularly preferred benefit agent is silicone, preferably silicones having viscosity greater than about 10,000 centipoise. The silicone may be a gum and/or it may be a mixture of silicones. One example is polydimethylsiloxane having viscosity of about 60,000

35 centistokes.

The benefit agent generally comprises about 10% to 40%, preferably 20% to 40%, most preferably 25% to 40% by weight of the chip composition.

#### Fumed Silica

Fumed silica is generally produced by the hydrolysis of silicon tetrachloride vapor in a flame of hydrogen and oxygen. The process produces particles of from about 7 to 30 millimicrons.

The enormous surface area and chain forming abilities are believed to allow it to form three-dimensional networks, altering flowing properties i.e., cause thickening.

Fumed silica will generally comprise the 0.01 to 10% by wt. of the composition, preferably 1% to 7% by wt., most preferably 1% to 5% by wt. of the composition.

#### Other Components

Water comprises 0 to 10%, preferably 0% to 8% by wt., most preferably 0.1 to 5% by wt. of the chip composition. It is sometimes preferred to have little or no additional water (other than that inherently present in the compounds) in the chip mixture because this may sometimes cause processing difficulties.

In addition the chip composition may comprise 0% to 15%, preferably 2% to 10% fatty acid, i.e.,  $C_8$  to  $C_{22}$  fatty acid. Generally, this is a straight chain, saturated fatty acid although this is not necessarily the case. The fatty acid helps to modify the wear rate of the emollient chip to better match that of the base soap.

### BAR COMPOSITIONS

In a second embodiment of the invention, the invention comprises extruded bar compositions in which 20% to about 40% of the chips used to make the final bars comprise the benefit agent additives (i.e. chips) described above and in which 80% to 60% of the chips comprise chips which comprise the surfactant system defining the final bar.

Specifically, the surfactant system chips comprise about 5% to 90% by wt. of a surfactant system wherein the surfactant is selected from the group consisting of soap (pure soap surfactant systems are included), anionic surfactant, nonionic surfactant, amphoteric zwitterionic surfactant, cationic surfactant and mixtures thereof. These chips may additionally comprise other components typically found in final bar compositions, for example, minor amounts of fragrance, preservative, skin feel polymer etc.

### Surfactant System

The term "soap" is used herein in its popular sense, i.e., the alkali metal or alkanol ammonium salts of aliphatic alkane- or alkene monocarboxylic acids. Sodium, potassium, mono-, di- and tri-ethanol ammonium cations, or combinations thereof, are suitable for purposes of this invention. In general, sodium soaps are used in the compositions of this invention, but from about 1% to about 25% of the soap may be potassium soaps. The soaps useful herein are the well known alkali metal salts of natural or synthetic aliphatic (alkanoic or alkenoic) acids having about 12 to 22 carbon atoms, preferably about 12 to about 18 carbon atoms. They may be described as alkali metal carboxylates of acrylic hydrocarbons having about 12 to about 22 carbon atoms.

Soaps having the fatty acid distribution of coconut oil may provide the lower end of the broad molecular weight range. Those soaps having the fatty acid distribution of peanut or rapeseed oil, or their hydrogenated derivatives, may provide the upper end of the broad molecular weight range.

It is preferred to use soaps having the fatty acid distribution of coconut oil or tallow, or mixtures thereof, since these are among the more readily available fats. The proportion of fatty acids having at least 12 carbon atoms in coconut oil soap is about 85%. This proportion will be greater when mixtures of coconut oil and fats such as tallow, palm oil, or non-tropical nut oils or fats are used, wherein the principle chain lengths are  $C_{16}$  and higher. Preferred soap for use in the compositions of this invention has at least about 85% fatty acids having about 12 to 18 carbon atoms.

Coconut oil employed for the soap may be substituted in whole or in part by other "high-alluric" oils, that is, oils or fats wherein at least 50% of the total fatty acids are composed of lauric or myristic acids and mixtures thereof. These oils are generally exemplified by the tropical nut oils of the coconut oil class. For instance, they include: palm kernel oil, babassu oil, ouricuri oil, tucum oil, cohune nut oil, murumuru oil, jaboty kernel oil, khakan kernel oil, dika nut oil, and ucuhuba butter.

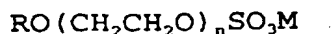
A preferred soap is a mixture of about 15% to about 20% coconut oil and about 80% to about 85% tallow. These mixtures contain about 95% fatty acids having about 12 to about 18 carbon atoms. The soap may be prepared from coconut oil, in which case the fatty acid content is about 85% of  $C_{12}$ - $C_{18}$  chain length.

The soaps may contain unsaturation in accordance with commercially acceptable standards. Excessive unsaturation is normally avoided.

5        Soaps may be made by the classic kettle boiling process or modern continuous soap manufacturing processes wherein natural fats and oils such as tallow or coconut oil or their equivalents are saponified with an alkali metal hydroxide using procedures well known to those skilled in the art.  
10       Alternatively, the soaps may be made by neutralizing fatty acids, such as lauric ( $C_{12}$ ), myristic ( $C_{14}$ ), palmitic ( $C_{16}$ ), or stearic ( $C_{18}$ ) acids with an alkali metal hydroxide or carbonate.

15       The anionic detergent active which may be used may be aliphatic sulfonates, such as a primary alkane (e.g.,  $C_8-C_{22}$ ) sulfonate, primary alkane (e.g.,  $C_8-C_{22}$ ) disulfonate,  $C_8-C_{22}$  alkene sulfonate,  $C_8-C_{22}$  hydroxyalkane sulfonate or alkyl glyceryl ether sulfonate (AGS); or aromatic sulfonates such  
20       as alkyl benzene sulfonate.

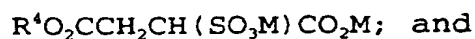
      The anionic may also be an alkyl sulfate (e.g.,  $C_{12}-C_{18}$  alkyl sulfate) or alkyl ether sulfate (including alkyl glyceryl ether sulfates). among the alkyl ether sulfates are  
25       those having the formula:



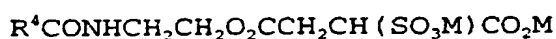
      wherein R is an alkyl or alkenyl having 8 to 18 carbons,  
30       preferably 12 to 18 carbons, n has an average value of greater than 1.0, preferably greater than 3; and M is a solubilizing cation such as sodium, potassium, ammonium or substituted ammonium. Ammonium and sodium lauryl ether sulfates are preferred.  
35

The anionic may also be alkyl sulfosuccinates (including mono- and dialkyl, e.g., C<sub>6</sub>-C<sub>22</sub> sulfosuccinates); alkyl and acyl taurates, alkyl and acyl sarcosinates, sulfoacetates, C<sub>8</sub>-C<sub>22</sub> alkyl phosphates and phosphates, alkyl phosphate esters and alkoxyl alkyl phosphate esters, acyl lactates, C<sub>8</sub>-C<sub>22</sub> monoalkyl succinates and maleates, sulphoacetates, alkyl glucosides and acyl isethionates.

Sulfosuccinates may be monoalkyl sulfosuccinates having the formula:

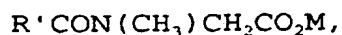


amide-MEA sulfosuccinates of the formula;



wherein R<sup>4</sup> ranges from C<sub>8</sub>-C<sub>22</sub> alkyl and M is a solubilizing cation.

Sarcosinates are generally indicated by the formula:



wherein R<sup>1</sup> ranges from C<sub>8</sub>-C<sub>20</sub> alkyl and M is a solubilizing cation.

Taurates are generally identified by formula:



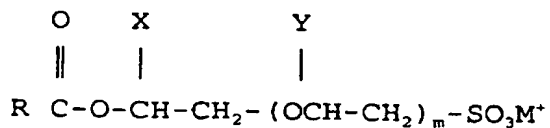
wherein R<sup>2</sup> ranges from C<sub>8</sub>-C<sub>20</sub> alkyl, R<sup>3</sup> ranges from C<sub>1</sub>-C<sub>4</sub> alkyl and M is a solubilizing cation.

Particularly preferred are the C<sub>8</sub>-C<sub>18</sub> acyl isethionates.

These esters are prepared by reaction between alkali metal isethionate with mixed aliphatic fatty acids having from 6 to 18 carbon atoms and an iodine value of less than 20. At least 75% of the mixed fatty acids have from 12 to 18 carbon atoms and up to 25% have from 6 to 10 carbon atoms.

Acyl isethionates, when present, will generally range from about 10% to about 70% by weight of the total bar composition. Preferably, this component is present from about 30% to about 60%.

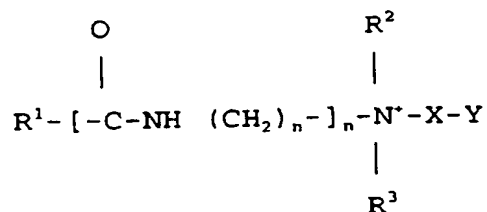
The acyl isethionate may be an alkoxyated isethionate such as is described in Ilardi et al., U.S. Patent No. 5,393,466, hereby incorporated by reference. This compound has the general formula:



wherein R is an alkyl group having 8 to 18 carbons, m is an integer from 1 to 4, X and Y are hydrogen or an alkyl group having 1 to 4 carbons and M<sup>+</sup> is a monovalent cation such as, for example, sodium, potassium or ammonium.

Amphoteric detergents which may be used in this invention include at least one acid group. This may be a carboxylic or a sulphonic acid group. They include quaternary nitrogen and therefore are quaternary amido acids. They should generally include an alkyl or alkenyl group of 7 to 18 carbon atoms. They will usually comply with an overall structural formula:

15



where R<sup>1</sup> is alkyl or alkenyl of 7 to 18 carbon atoms;

R<sup>2</sup> and R<sup>3</sup> are each independently alkyl, hydroxyalkyl or carboxyalkyl of 1 to 3 carbon atoms;

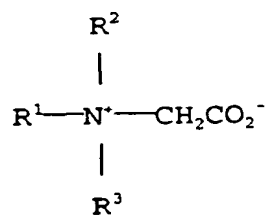
m is 2 to 4;

n is 0 to 1;

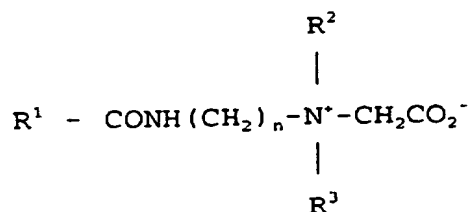
x is alkylene of 1 to 3 carbon atoms optionally substituted with hydroxyl, and

Y is -CO<sub>2</sub>- or -SO<sub>3</sub>-

Suitable amphoteric detergents within the above general formula include simple betaines of formula:



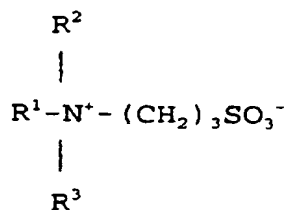
and amido betaines of formula:



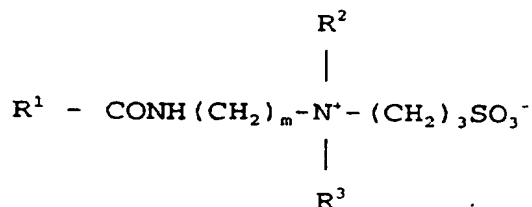
where n is 2 or 3.

In both formulae  $R^1$ ,  $R^2$  and  $R^3$  are as defined previously.  $R^1$  may in particular be a mixture of  $C_{12}$  and  $C_{14}$  alkyl groups derived from coconut so that at least half, preferably at least three quarters of the groups  $R^1$  have 10 to 14 carbon atoms.  $R^2$  and  $R^3$  are preferably methyl.

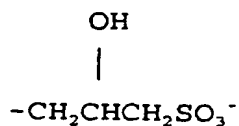
A further possibility is that the amphoteric detergent is a sulphobetaine of formula:



or



where m is 2 or 3, or variants of these in which  $-(CH_2)_3SO_3^-$  is replaced by



In these formulae  $R^1$ ,  $R^2$  and  $R^3$  are as discussed previously.

5 The nonionic which may be used as the second component of the invention include in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example aliphatic alcohols, acids, amides or alkylphenols with alkylene oxides, especially ethylene  
10 oxide either alone or with propylene oxide. Specific nonionic detergent compounds are alkyl ( $C_6-C_{22}$ ) phenols ethylene oxide condensates, the condensation products of aliphatic ( $C_8-C_{18}$ ) primary or secondary linear or branched  
15 alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylenediamine. Other so-called nonionic detergent compounds include long chain tertiary amine oxides, long chain tertiary phosphine oxides and dialkyl sulphoxides.

20 The nonionic may also be a sugar amide, such as a polysaccharide amide. j Specifically, the surfactant may be one of the lactobionamides described in U.S. Patent No. 5,389,279 to Au et al. which is hereby incorporated by reference or it may be one of the sugar amides described in Patent Nol 5,009,814 to Kelkenberg, hereby incorporated into the subject application by reference.

25 Examples of cationic detergents are the quaternary ammonium compounds such as alkyldimethylammonium halogenides.

30 Other surfactants which may be used are described in U.S. Patent No. 3,723,325 to Parran Jr. and "Surface Active Agents and Detergents" (Vol. I & II) by Schwartz, Perry & Berch, both of which are also incorporated into the subject application by reference.

35 Although the bar may be a pure soap bar, preferably the surfactant system of this chip (forming the surfactant system

in the bar) comprises:

- (a) a first synthetic surfactant which is anionic; and
- (b) a second synthetic surfactant selected from the group consisting of a second anionic different from the first, a nonionic, an amphoteric and mixtures thereof.

The first anionic can be any of those recited above, but is preferably a C<sub>8</sub> to C<sub>18</sub> isethionate as discussed above. Preferably acyl isethionate will comprise 10% to 90% by wt. total bar composition.

The second surfactant is preferably a sulfosuccinate, a betaine or mixtures of the two. The second surfactant or mixture of surfactant will generally comprise 1% to 10% total bar composition. A particularly preferred composition comprises enough sulfosuccinate to form 3-8% total bar compositions and enough betaine to form 1-5% of total bar composition.

### Processing

In general, the additive, benefit agent chips are formed by mixing the ingredients in a mixer at a temperature just above the melting point of the polyalkylene glycol (e.g., about 50°C and above, generally no higher than about 110°C) for about 1 to 60 minutes, and then cooling in a chill roll. Order of addition is not critical. The "non" benefit agent chips are formed by similarly mixing and cooling.

The chips are then combined, for example, in a hopper or ribbon mixer where they may be refined (e.g., worked into a more pliable mass), plodded into billets, stamped and cut.

In a third embodiment of the invention, the invention relates to a method of forming additives (chips) containing a benefit agent which method comprises:

- (a) mixing polyalkylene glycol, benefit agent, fumed silica, optional water and optional fatty acid in a container for 1 to 60 minutes at about above 50°C; and
- (b) cooling the mixture on a chill roll to about 0 to 25°C to form chips.

The following examples are intended to further illustrate the invention and are not intended to limit the invention in any way.

Unless stated otherwise, all percentages are intended to be percentages by weight.

### EXAMPLES

#### Protocol

Silicone measurement was conducted as follows:

Analysis is done by method known as ICP (Inductively Coupled Argon Plasma). This procedure required a step involving extraction with xylene, and is therefore currently used only in-vitro. The ICP technique employed a Thermo Jarrell Ash Atom Scan 25 with measurements being made at 251.612 nm. Additional ICP measurement parameters are given below.

The treatment process was as follows:

The porcine skin was shaved, dermatomed, and sectioned into 25 cm pieces prior to treatment. The skin sample was

then treated by rubbing the bar sample across the skin 10 times, in a back and forth motion. The resulting liquor on the skin was lathered for 30 seconds and then rinsed for 10 seconds with water which was regulated at 90-95°F. The treated skin sample was placed in a borosilicate scintillation vial that contained 10 ml of xylene. The samples were placed on a platform shaker for 1 hour to allow for the extraction of the silicone. After the extraction period, the skin was removed from the vial and the extract was analyzed using ICP technique. Sample solutions were tested against a 10 pm silicone standard.

Typical ICP Measurement Parameters for Measuring Silicone in Xylene

Torch gas flow	high
Auxiliary gas flow	1.5 L/min
Analyzer pump rate	0.9 m L/min
Nebulizer pressure	21 psi
Observation height	12 mm above load cell
Plasma power	1750 W
Wavelength	251.612 nm
Slit height	6 mm
Integration time	4 sec

Example 1

Using the protocol discussed above, benefit agent deposition (e.g., deposition of silicone) was measured in compositions representing (1) the bar of Visscher et al. with no fumed silica chips; (2) the bars of the invention which did contain fumed silica chips; and (3) a liquid body wash composition. Each is discussed in greater detail below:

(1) Visscher Bar (WO 92/08444)

The Visscher bar was obtained following the procedure taken from WO 92/08444 (equivalent to U.S. Patent No. 5,154,849) where polyethylene glycol is used as a carrier for silicone in bars (procedure was done in a Patterson mixture). Procedure was as follows:

(a) 681 gm of Carbowax PEG 8000 was melted and held around 60°C;

(b) 400 gm of GE 350 cps silicone was added; and

(c) 273 gm of GE 500,000 cps silicone was added.

(The patent explains the carrier to be 10:9 silicone A:PEG where silicone A is a blend of 40:60 silicone gum, 500,000 cps to silicone fluid, 350 cps)

The mixture remained in the mixer for 45 minutes until it was considered homogenous. The mixture was then removed and placed on a chill roll set at 7°C. The resulting "chips" were soft, pliable and severely tacky. Silicone covered the entire surface of the equipment.

A sample bar was prepared by chip mixing surfactant chip: Visscher chip ratio of 4:1 (wherein surfactant chip comprises 40-60% fatty acid isethionate, 20-30% fatty acid, 1-10% sodium isethionate, 1-10% sulfosuccinate, about 5% betaine, preservatives, dyes and minors); and extruding into a billet with a Weber Selander plodder. The resulting billet was soft and from experience not considered a viable product. The pressed bar lathered poorly. From experience this type of "chip" cannot be produced using conventional equipment.

More specifically, mixing surfactant chips and Visscher chips at a weight ratio of 4:1, respectively, resulted in

large, non-free flowing clumps which adhered together by surface silicone. This result impeded feeding into the extruder. Material which did feed was extruded as a soft, sticky billet. When stamped, the bar had a poor surface, was tacky and produced little lather when wetted.

(2) Bar of the Invention

The bar of the invention comprised a 70%/30% mixture of chips wherein the 30% additive chip component had the following formulation range:

40-100%, preferably 40-80% polyethyleneglycol (e.g. PEG 8000);

10-50%, preferably 10-40% polydimethyl siloxane of 60,000 centistokes;

0.1 to 10%, preferably 1 to 5% Cab-o-sil<sup>®</sup> fumed silica (e.g., fumed silica 45-5);

0-20%, preferably 1-10% deionized water; and

0-20%, preferably 0-10% to C<sub>8</sub> to C<sub>22</sub> fatty acid

and

the 70% surfactant chips were like the surfactant chips used in the Visscher et al. bar, as follows:

about 40-60% by wt. fatty acid isethionate;

about 20-30% by wt. fatty acid;

about 1-10% by wt. sodium isethionate

about 1-10% by wt. sulfosuccinate;

about 5% by wt. betaine; and

remainder preservative, dyes, water and other minors.

A preferred benefit agent chip comprises as follows:

(a) 55-65% PEG

(b) 25-40% silicone

(c) 1-7% silica; and

(d) 0-8% deionized water.

The chips were mixed, plodded together at the above-identified ratios, and extruded into bars.

(3) Liquid Body Wash

The liquid body wash had the following formulation:

	% by wt.
Betaine	5-15%
Sodium Cocoyl Isethionate	1-10%
Anionic	1-5%
Fragrance, preservatives	0.1-2.0%
Water	to balance

As noted deposition results were taken using the ICP techniques discussed and results set forth as follows:

	Deposition.
Visscher Bar	2.16 +/- 0.48 $\mu\text{g}/\text{cm}^2$
Bar of Invention	2.24 +/- 0.83 $\mu\text{g}/\text{cm}^2$
Liquid	2.14 +/- 0.62 $\mu\text{g}/\text{cm}^2$

It is surprising that the bar can deposit as well as the liquids. Moreover, in contrast to Visscher, the bar of the invention was readily processable and did not clog machinery (See Example 2).

Example 2

To further show differences between the bar of the invention and bars of Visscher, applicants decided to analyze the chips more closely.

Chips used in formation of the Visscher et al. bar, and chips carrying benefit agent and used in the formation of the bars of the invention were micrographed.

5       As seen from Figures 1 and 2, the Visscher et al. (P&G) chips show large "blobs" of silicone surrounding the alkylene glycol while the chips of the invention show small discrete droplets of silicone.

10       While not wishing to be bound by theory, it is believed the difference in amount of silicone and how it is formed accounts for the tremendous processing difficulties experienced in forming the P&G bars relative to those of the invention. As  
15       noted above, 4:1 ratio of Visscher chips to surfactant chips formed large non-free flowing clumps which hindered chip feeding into the extruder and noodle processing. The clumps  
20       also caused agglomeration in the vacuum chamber which significantly reduced billet formation. Further, as noted, material which did extrude was soft and sticky and, when stamped, the bar had a poor surface, was tacky and produced little lather when wetted.

CLAIMS

1. A soap chip composition comprising:

- 5 (a) 40% to about 80% by wt. of chip composition alkylene glycol having a molecular weight from about 4,000 to 100,000;
- (b) 10% to 40% by wt. chip composition benefit agent;
- (c) 0.01 to 10% by wt. chip composition fumed silica;
- (d) 0 to 10% by wt. chip composition water; and
- 10 (e) 0% to 15% by wt. chip composition C<sub>8</sub> to C<sub>22</sub> fatty acid.

2. A composition as claimed in claim 1, wherein MW is 5,000 to 10,000.

15 3. A composition as claimed in claim 1 or claim 2, wherein said polyalkylene glycol is propylene glycol.

4. A composition as claimed in any preceding claim, wherein said benefit agent is silicone.

20 5. An extruded toilet bar composition comprising 20-40% chips comprising:

- 25 (a) 40% to about 80% by wt. of chip composition polyalkylene glycol have a molecular weight from about 4,000 to 20,000;
- (b) 10% to 40% by wt. chip composition benefit agent;
- (c) 0.01 to 10% by wt. chip composition fumed silica;
- (d) 0 to 10% by wt. chip composition water; and
- 30 (e) 0% to 15% by wt. chip composition C<sub>8</sub> to C<sub>22</sub> fatty acid, and 80% to 60% chips comprising 5% to 90% of a surfactant system wherein the surfactant is selected from the group consisting of soap, anionic surfactant, nonionic surfactant, amphoteric surfactant, cationic surfactant and mixtures thereof;

6. A composition as claimed in claim 5, wherein said surfactant system comprises

- (a) a first anionic surfactant; and
- (b) a second surfactant selected from the group consisting of a second anionic different from the first, a nonionic, an amphoteric and mixtures thereof.

7. A composition as claimed in claim 6, wherein the first anionic is acyl isethionate; optionally, wherein the isethionate forms 10% to 70% of the final bar composition.

8. A composition as claimed in claim 6, wherein the second surfactant is either (i) sulfosuccinate or (ii) betaine, optionally wherein the betaine is amidococoylbetaine; or (iii) a mixture of sulfosuccinate and betaine.

9. A process of forming a chip comprising:

- (a) 40% to about 80% by wt. of chip composition alkylene glycol have a molecular weight from about 4,000 to 20,000;
- (b) 10% to 40% by wt. chip composition benefit agent;
- (c) 0.01 to 10% by wt. chip composition fumed silica;
- (d) 0 to 10% by wt. chip composition water; and
- (e) 0% to 15% by wt. chip composition C<sub>8</sub> to C<sub>22</sub> fatty acid.

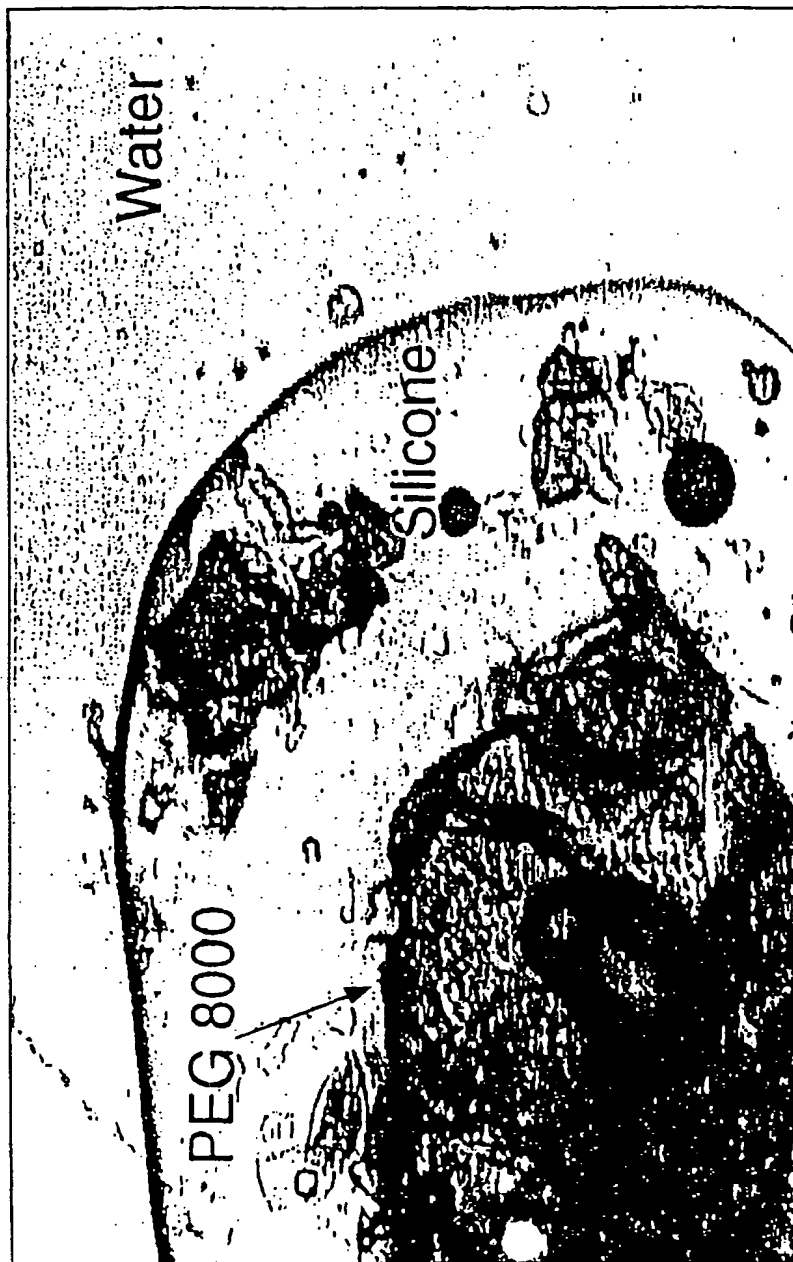
wherein said process comprises mixing the ingredients of subparagraph (a) - (e) for 1 to 60 minutes at a temperature of about above 50°C; and then cooling said mixture on a chill roll at a temperature of 0° to 25°C.

10. A method of enhancing deposition of benefit agent from a bar composition without compromising processing which method comprises using in said bar compositions about 20% to 40% chips comprising:

- (a) 40% to about 80% by wt. of chip composition polyalkylene glycol have a molecular weight from about 4,000 to 100,000;
- (b) 10% to 40% by wt. chip composition benefit agent;
- (c) 0.01 to 10% by wt. chip composition fumed silica;
- (d) 0 to 10% by wt. chip composition water; and
- (e) 0% to 15% by wt. chip composition C<sub>8</sub> to C<sub>22</sub> fatty acid.

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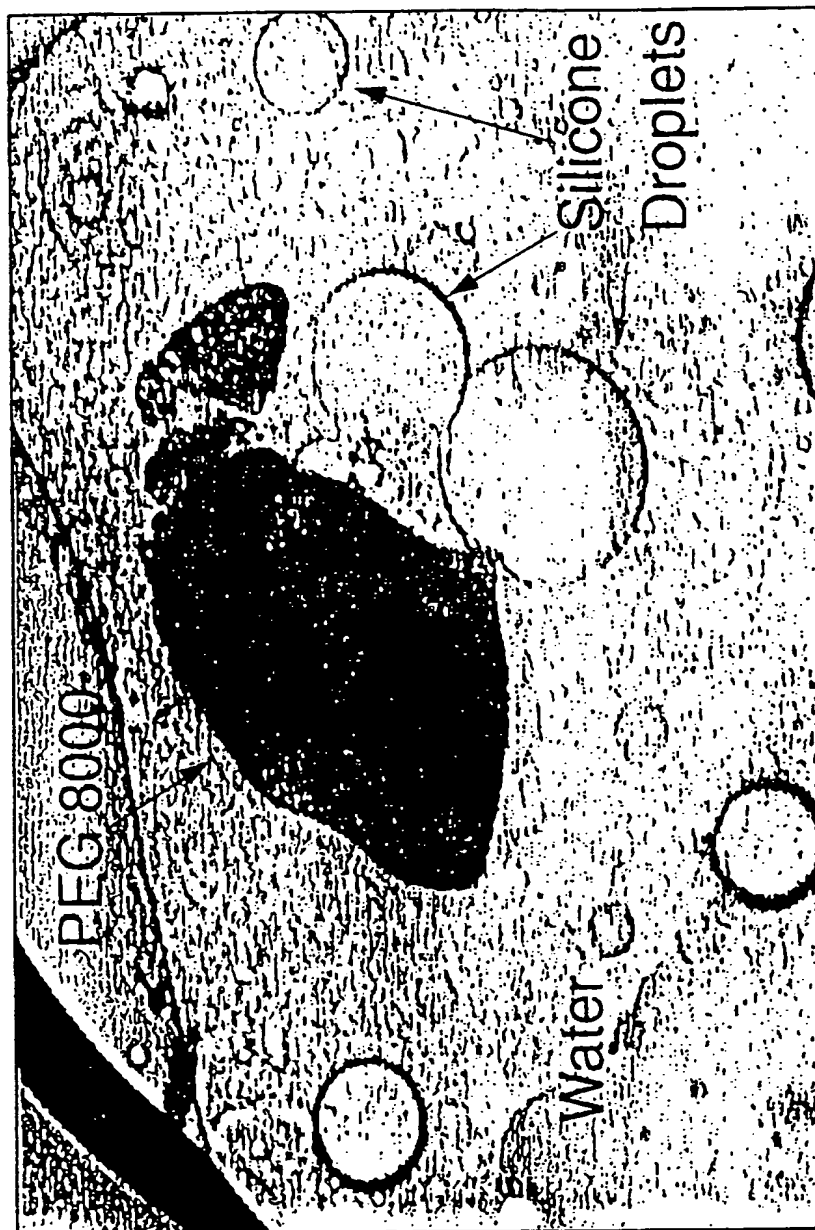
Fig.1. P & G Chip  
(Unprocessable)



▪ Silicone coats PEG chip.

2/2

Fig.2.  
URUS Chip  
(Processable)



- PEG traps PDMS as droplets upon crystallisation
- PEG chip goes into solution when flooded.
- Individual PDMS droplets released

## INTERNATIONAL SEARCH REPORT

International Application No  
PCT/EP 97/00896

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 A61K7/48 C11D17/00 A61K7/50

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 A61K C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 92 08444 A (PROCTER & GAMBLE) 29 May 1992 see claims 1-5 & US 5 154 849 A cited in the application ---	5-8
A	WO 94 17172 A (UNILEVER PLC ;UNILEVER NV (NL)) 4 August 1994 see page 4, line 1 - page 16, line 20 ---	1,4-9
A	EP 0 294 206 A (UNILEVER PLC ;UNILEVER NV (NL)) 7 December 1988 ---	
P,A	WO 96 28140 A (PROCTER & GAMBLE) 19 September 1996 -----	



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

## \* Special categories of cited documents:

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

22 August 1997

Date of mailing of the international search report

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

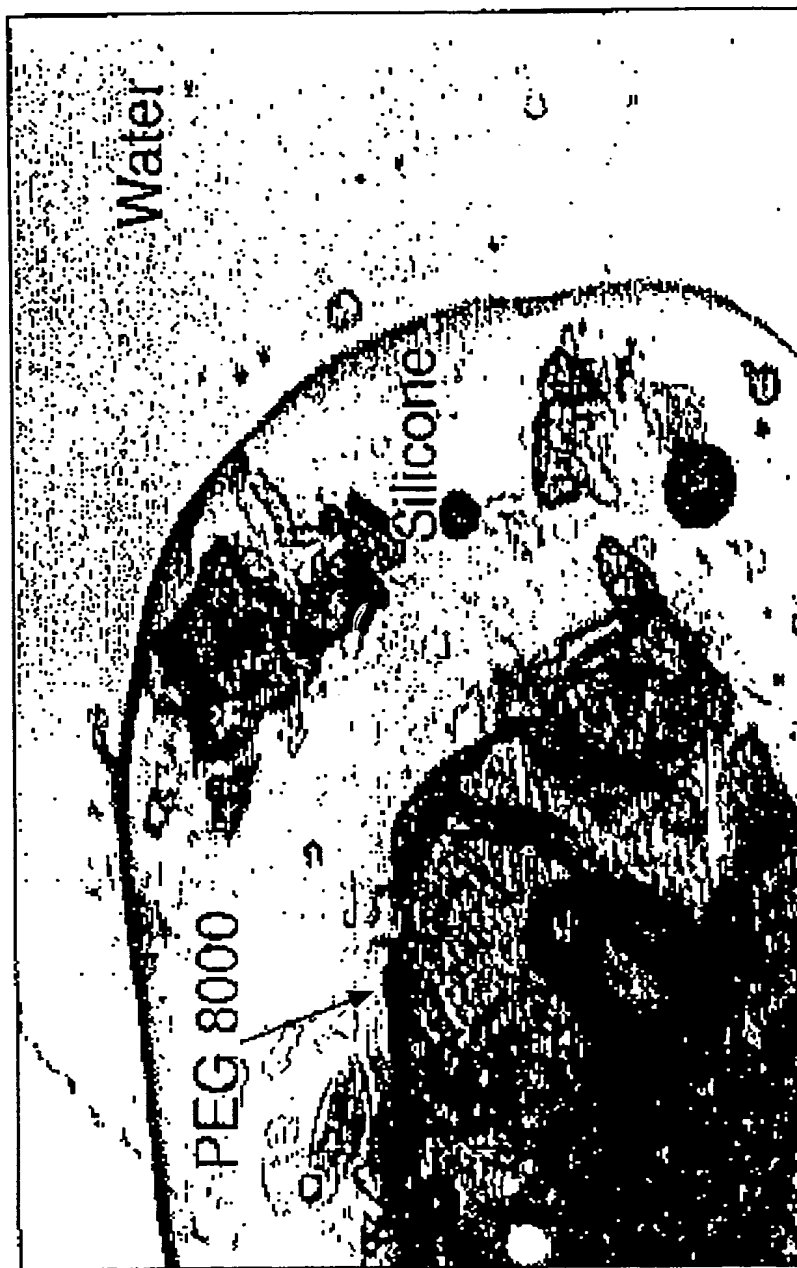
PCT/EP 97/00896

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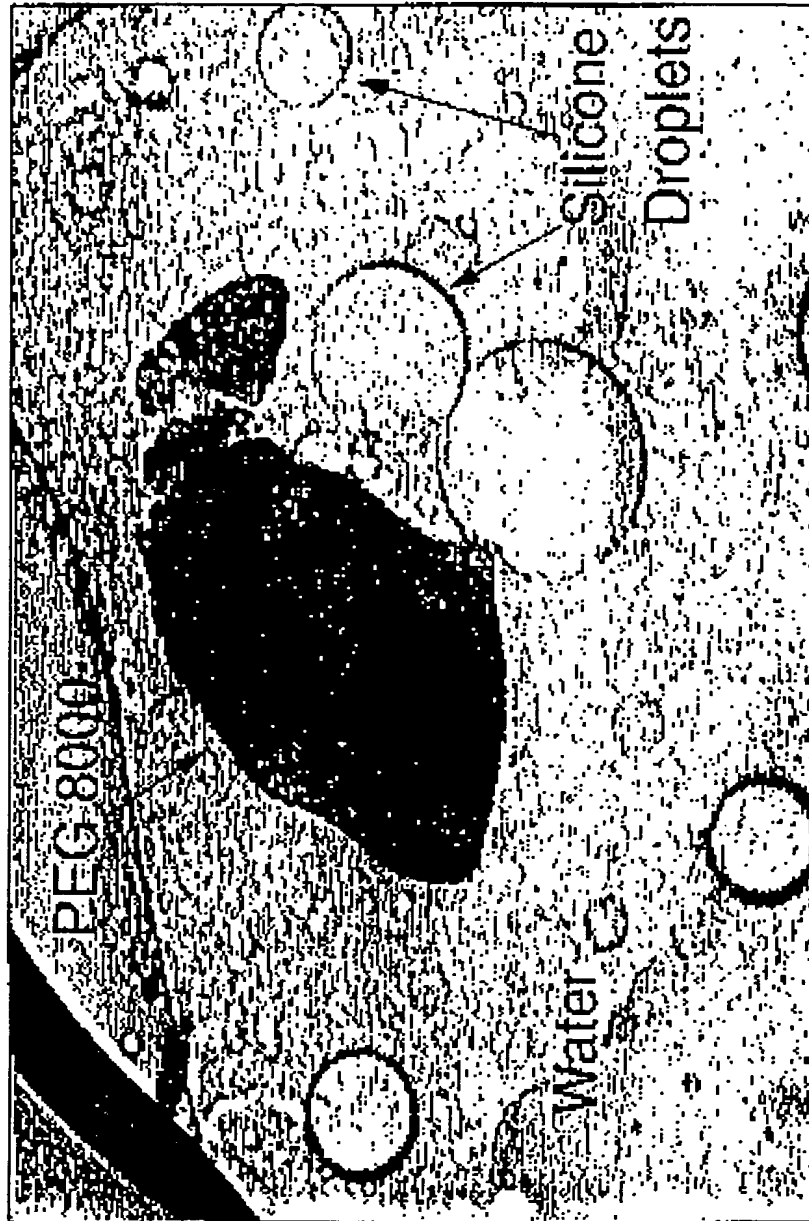
Fig.1. P & G Chip  
(Unprocessable)



■ Silicone coats PEG chip.

2/2

Fig.2.  
URUS Chip  
(Processable)



- PEG traps PDMS as droplets upon crystallisation
- PEG chip goes into solution when flooded.
- Individual PDMS droplets released